

INDUSTRIAL HYGIENE SAMPLING GUIDE FOR CONSOLIDATED INDUSTRIAL HYGIENE LABORATORIES (CIHLs)

Prepared by

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(CIHLs)

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INTRODUCTION

This guide contains a compilation of sampling and analytical method recommendations for specific chemicals which the Navy has in-house analytical capability through its three Consolidated Industrial Hygiene Laboratories (CIHLs) located at the Navy Environmental and Preventive Medicine Unit Two (NAVENPVNTMEDU TWO), Norfolk, VA; NAVENPVNTMEDU FIVE, San Diego, CA and NAVENPVNTMEDU SIX, Pearl Harbor, HI. This guide is a concise reference for the industrial hygienist in the proper submission of industrial hygiene, environmental, bulk and biological samples. This guide lists the analyte or substance, the Chemical Abstracts Service Registry Number (CAS #) for the substance, analytical method used by the laboratory in performing the analysis, method's coefficient of variation (CV), limit of detection (LOD), sampling media, recommended air volume, sampling rate, special instructions for the industrial hygienist submitting the sample, and location of laboratory which can analyze the sample. Customers should submit samples to the laboratory located nearest them or most convenient to them. If that laboratory does not have the analytical capability, call the laboratory to verify this fact and choose a laboratory which provides the required service. Since all laboratories are constantly updating their analytical services, always check with the closest laboratory first.

Each CIHL welcomes comments and suggestions regarding its services, additional method development requirements, alternate sampling techniques, and any other input. All questions regarding laboratory service/capability should be addressed to the CIHL which provides the service. Working hours are generally 0730 to 1600 hours Monday through Friday. If the CIHL can't be reached or additional information is required, please call the NAVENPVNTMEDU to which the laboratory reports. All comments concerning the CIHL program management, additions, corrections and changes to this guide, should be addressed to:

Commanding Officer
Attn: CIHL Program Manager
Navy Environmental Health Center
2510 Walmer Avenue
Norfolk, VA 23513-2617

Tel: (757) 462-5526
DSN: 253-5526
FAX: (757) 445-7330
<http://www-nehc.med.navy.mil/>

LABORATORY ORGANIZATION

BUMEDINST 5450.157, dated 15 June 92, published the functions and tasks of the Navy Environmental Health Center (NAVENVIRHLTHCEN) and its subordinate commands. The mission of the NAVENVIRHLTHCEN, to coordinate and provide centralized support and services to medical activities, afloat and ashore, in areas of occupational health, lead to the formation of the CIHL program. BUMEDINST 5450.157 requires the NAVENPVNTMEDUs to provide, through the CIHLs, specialized qualitative and quantitative analyses of samples to support occupational health and industrial hygiene investigations and assessments.

On 1 October 1989, all Consolidated Industrial Hygiene Laboratories became Departments within the Navy Environmental Health Center's Echelon IV commands. The following information for the three CIHLs is provided:

1- Officer In Charge

Navy Environmental Preventive Medicine Unit Five (NAVENPVNTMEDU FIVE)
Consolidated Industrial Hygiene Laboratory (CIHL)
Naval Station, Box 368143
3235 Albacore Alley
San Diego, CA 92136-5199

Mr. Charles Kubrock, Director of Laboratory
Phone: (619) 556-7070 DSN: 526-7070
FAX: (619) 556-1492
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NAVENPVNTMEDU FIVE Phone: (619) 556-7070
DSN: 526-7070 FAX: (619) 556-7071

2- Officer In Charge

Navy Environmental Preventive Medicine Unit Six (NAVENPVNTMEDU SIX)
Consolidated Industrial Hygiene Laboratory (CIHL)
1215 North Road
Pearl Harbor, HI 96860-4477

Dr. Roy M. Ishikawa, CIH, Director of Laboratory
Phone: (808) 474-4428; DSN: 474-4428
FAX: (808) 474-2071
Email: cihl@nepmu6.med.navy.mil
<http://nepmu6.med.navy.mil/>

NAVENPVNTMEDU SIX Phone: (808) 473-0555
DSN: 473-0555 FAX: (808) 473-2754

3- Officer In Charge

Navy Environmental Preventive Medicine Unit Two (NAVENPVNTMEDU TWO)
Consolidated Industrial Hygiene Laboratory (CIHL)
1887 Powhatan Street
Norfolk, VA 23511-3394

Mr. George Lindsay, CIH, Director of Laboratory

Phone: (757) 444-7671 x 3038; DSN: 564-7671

FAX: (757) 444-1556

E-mail: lindsayg@nepmu2.med.navy.mil

<http://www-nehc.med.navy.mil/nepmu2/>

NAVENPVNTMEDU TWO Phone: (Same as lab)

GENERAL POLICY

The CIHLs provide analytical support services for samples submitted through the BUMED Industrial Hygiene Offices. The analytical services available at the laboratories are primarily designed for quantitative analyses of occupational health samples and selective environmental samples.

SPECIFIC POLICIES

POLICY ON STANDARD OPERATING PROCEDURES AND LOCAL OPERATING PROCEDURES

Standardization among the laboratories is an essential part of the CIHL programs . A document of Standard Operating Procedures (SOPs), dated June 93, issues quality guidance for the operation and standardization among the CIHLs. Based on this document each CIHL developed its own Local Operating Procedures (LOPs). The LOP (which implements instructions and any laboratory procedural changes) maintains current procedures in use at each laboratory. Historical records are kept of the dates when procedures are implemented and taken out of service.

POLICY ON SAMPLE ACCEPTANCE/REJECTION

Sample submissions, accompanied by the completion of Forms NEHC 5100/13 or 5100/14 (Note: every information category must be completed) by the industrial hygienist, properly preserved as appropriate, and shipped by the proper method (See Sample Packaging and Shipping Requirements Section) will be accepted by the CIHL and analyzed as routine unless the submission is noted "URGENT". Urgent sample must arrive by a one or two day courier service.

If samples are taken incorrectly and/or incomplete paperwork is received, every effort will be made to secure the necessary information to convert the invalid sample into a valid sample. Samples will only be returned to the client when requested by the client. Paperwork may be returned for correct completion, however the samples will remain at the laboratory.

In order to assure a quick laboratory turnaround time, please assure samples are taken according to this guide, shipped appropriately, and the submission Forms are correct and complete.

When samples are received and are not able to be corrected for validity (e.g., fiber counts submitted on PVC filters), the client will be notified by phone, fax or letter in order to determine the disposition of the sample. These samples will be returned to the customer upon the request of the customer.

POLICY ON ANALYTICAL METHODS

Rarely are analytical methods either complete or fully comprehensive to preclude some interpretation, change or modification of the method. NOTE: This is the reason for the CIHL requirement that a LOP manual be available at each CIHL. Most methods are single analyte methods while most samples are multiple contaminants. Most analytical methods used by the CIHLs are taken from the National Institute for Occupational Health (NIOSH) or the Occupational Safety and Health Administration (OSHA) methods. Since OSHA does not required specific analytical methods, unless stated in stressor-specific standards, any method (e.g., ASTM, literature, journal articles, etc.) can be used as long as it meets NIOSH criteria of accuracy within 25% at the 95% confidence level. All NIOSH and OSHA methods in this document are "modified methods". The modification is necessary because of the variance in: analytical columns (types, sizes); desorbing agents; digesting acids/bases; analytical equipment conditions (temperatures, pressures, flow rates). All these modified methods are evaluated and validated for the NIOSH accuracy of 25% at the 95% confidence level by each CIHL, and the method changes are documented as modifications.

POLICY ON COEFFICIENTS OF VARIATIONS

Randomly distributed errors occurring in industrial hygiene sampling are normal and are commonly included in analytical reports by the use of the coefficient of variation (CV). The CV is a useful index of differentiating the true mean of known data points and laboratory reported data. The total CV of the sampling and analytical method is based on a statistical standard normal deviation for 95% two-sided confidence limits. The statistical decision techniques developed by NIOSH and OSHA are implemented in the Navy's use of the CVs. Therefore since the industrial hygienists will seldom receive true exposure results from the labs due to sampling and analytical fluctuations, the CVs for each analyte are reported in the tables so 95% confidence levels may be calculated. Our CIHLs are capable of reporting the same or lower CVs annotated in the official analytical method (the values of which are noted in the Laboratory Sampling Guide Table).

For Time Weighted Average (TWA) sampling, the CV criteria originally adopted by NIOSH of $\pm 25\%$ accuracy, with 95% confidence limits, is usually cited, but accuracy specifications may vary from one standard to the next. Substances which have Permissible Exposure Levels (PELs), but for which no specific standard has been promulgated, do not have specific accuracy requirements. For these substances, the labs consider the method acceptable (e.g., OSHA, NIOSH, literature cited methods) if it can meet the 25% accuracy requirement with 95% confidence.

POLICY ON ANALYTICAL RESULTS

The CIHLs are reporting air samples results in "total weight per sample" because of the confusion in the interpretation when samples are reported in mg/M^3 (Some clients are using this value as the TWA when the sample did not represent an eight-hour exposure.) Blanks submitted with the samples are also reported in "total weight per sample". The CIHL will notify the client when the blank values are elevated more than normal. It is now the responsibility of the client to take the analytical results and compute TWAs as necessary. If you need assistance, please contact your local industrial hygienist or the CIHL.

POLICY ON LIMIT OF DETECTION

It is not unusual for the Limit of Detection (LOD) of an analyte to vary from day to day. Instrumental conditions and environments vary day to day and this variation often effects the LOD. If you envision detection levels (e.g., a short duration sample) to be a problem, please contact the CIHL performing the analyses. Often the laboratory can modify a method to increase the sensitivity and selectivity; however, the analyst must know your requirements before the performance of the analyses using the standard analytical method.

POLICY ON SPIKED SAMPLES/FIELD SUBMITTED QC SAMPLES

The CIHLs are required by their accreditation through the American Industrial Hygiene Association (AIHA) to have a comprehensive quality control (QC) program which involves, at a minimum: A written QC plan, a designated Quality Control Coordinator (QCC) responsible for the QC program, participation in the Proficiency Analytical Testing (PAT) program for all categories of analytes performed for the client, records which demonstrate the routine introduction of control samples of known content along with samples for analysis, records which demonstrate routine checks, calibrations, maintenance of equipment and instruments are performed to ensure adequate performance, quality control data stored in an accessible manner, routine checks made of procedures and reagents, and interlaboratory, as well as intralaboratory QC.

Occasionally the client may feel uncomfortable with laboratory results and thereby "test" the QC program of the laboratory by administering blind QC samples to the laboratory.

The only recommended method of testing the laboratory is purchasing past PAT rounds from the

AIHA and submitting these as controlled spikes. Literature articles have proven that side by side duplicate monitoring very rarely produces duplicate samples. The use of a duplicate sampling manifold will not produce duplicate samples; however this method of sampling is superior to the use of two independent sampling systems side by side. Contact the AIHA (phone number (703) 849-8888 or FAX (703) 207-3561) for the purchase of PAT metals, solvents, fibers and free silica. PLEASE NOTIFY THE LABORATORY ONCE YOU RECEIVE THE RESULTS OF YOUR QC SAMPLE SO THE LABORATORY MAY DOCUMENT ITS QUALITY CONTROL PROGRAM TO INCLUDE THIS BLIND QC SAMPLE. THIS SAMPLE WILL BE IDENTIFIED AS A TRUE QC BLIND. Also the laboratory will remove the results of this sample from the Database if you have identified the sample as a field sample (e.g., assigned fictitious breathing zone sample information).

Please realize that if there is a quality problem with the CIHLs, the labs want to be the first to know so they can find and resolve the problem. The labs welcome and expect feedback from the clients.

POLICY ON BLANK MEDIA

NIOSH recommends and it is the CIHLs policy that 1) field blanks* should be treated like field samples (corrected for reagent blanks**, media blanks*** and recovery) and 2) sample concentration correction from contaminated field blanks be performed by the person submitting the sample, and NOT THE LABORATORY. (This NIOSH recommendation directed the CIHLs to report all samples and blanks in total contaminants per sample). It is the responsibility of the client (with assistance from the laboratory) to determine what constitutes a contaminated field blank, whether the field blank should be subtracted from the associated sample and whether the sample result should be accepted as valid.

* Field blanks measure the signal contribution of the media plus any contamination which may have occurred during handling, shipping and storage before analysis. Field blanks are taken into the field (workplace), opened and treated just like the field samples except no air is drawn through them.

** Reagent blanks are virgin sampling media that have not been in the workplace.

*** Media blanks measure the signal contribution from the collection media (e.g., the filter, sorbent tube, the impinger solution, etc.)

The nature and number of blank measurements will depend on the method and circumstances. The purpose of all blank measurements is to help prevent errors in identification and quantifying the field samples.

Other CIHL policies on blanks include:

1. All requests for sample analysis will require a minimum of one field blank when OSHA sampling and analytical methods are used, or a minimum of two field blanks when NIOSH sampling and analytical methods are used.

2. When multiple requests from the same source and for the same type of analysis (e.g., metals, benzene, Cr VI, etc.) with multiple corresponding blanks are submitted, the laboratory will analyze and report each sample and each blank submitted.
3. Blanks are analyzed as samples and reported as amount of analyte per sample (e.g., 2 milligrams Cd per filter) or as less than the limit of detection (i.e., <LOD). The LOD appears on the report and will be equal to, or approximately equal to, the analytical method's LOD (See column LOD in the Lab Sampling Guide). It is not unusual for the Limit of Detection (LOD) of an analyte to vary from day to day. Instrumental conditions and environments vary day to day and this variation often effects the LOD. If you envision problems with published detection levels (e.g., a short duration sample), please contact the CIHL performing the analyses. Often the laboratory can modify a method to increase sensitivity or selectivity; however, the analyst must know your requirements before the performance of the analyses using the standard analytical methods.
4. In your computation of sample results, sample values are corrected by subtracting the blank value (or average of blank values when more than one blank is submitted) from each sample value.
5. High blanks - Definition. The majority of blanks for gas chromatographic analyses and metal analyses yield values that are below the LOD for the chosen analyte. For those analytes any blank value which exceeds the LOD can be considered "high". For certain metals (e.g., Fe and Zn), solvents (e.g., benzene) and other analytes (e.g., sulfate) the blank values may exceed the LOD. For these analytes, the lab calculates the mean and the standard deviation of ten or more blanks previously analyzed and uses the mean plus 3 times the standard deviation as the control limit. Any blank which exceeds this control limit will be considered "high". The CIHL will inform you when high blanks are encountered. NOTE: The AIHA considers high blank values to be samples where the reported value (e.g., x micrograms) divided by the volume of sample collected (e.g., y liters) exceeds one-tenth the PEL. This type of contamination may occur in manufacturing or handling of the samples. If possible, additional blanks should be analyzed to ensure sampling medium lot integrity. The industrial hygienist should work closely with the chemists in the CIHLs to determine the source of contamination and to determine whether it is constant. One approach is to decide whether all the samples collected are contaminated equally. If there is a consistent source of contamination, the sample results should be at or above the blank level. If there is no consistency, some unexplained phenomenon has occurred, and the blank should not be subtracted from the analytical results. Bear in mind that subtracting the blank may reduce sample results to lower than what is truly in the workplace. It is generally preferable to be conservative by slightly overestimating the exposures rather than underestimating. It may be useful for the industrial hygienist to prepare control charts for blank values for each type of sample (e.g., lead, zinc, toluene, etc.) in order to determine an acceptable blank value range.

POLICY ON USE OF DISCLAIMERS

The CIHLs recognize field situations when samples cannot be taken according to required sampling methods (e.g., "a once in a lifetime opportunity sample"). In such cases, the laboratory

will usually analyze the sample if taken on an appropriate sampling medium, and report a result accompanied by a disclaimer STAMP. The more common disclaimers on the stamp are:

- 1- INSUFFICIENT AIR VOLUME- The air volume is less than the amount recommended for this method. Consequently the coefficient of variation (CV) published for the method may not apply. Professional judgement should be used with the interpretation of results.
- 2- QUESTIONABLE FLOW RATE- The flow rate differs from the recommended method's rate. Therefore, professional judgement should be used in the interpretation of results.
- 3- INCORRECT SAMPLING MEDIUM- The sample media is not one currently recommended by NIOSH, OSHA or the latest edition of NAVENVIRHLTHCEN's Industrial Hygiene Sampling Guide for CIHLs. Therefore, professional judgment must be used in the interpretation of results.
- 4- NON-NIOSH/NON-OSHA METHOD- The analytical method is not one currently recommended by NIOSH, OSHA or the latest edition of NAVENVIRHLTHCEN's Industrial Hygiene Sampling Guide for CIHLs. Therefore, professional judgment must be used in the interpretation of results.
- 5- SHIPPING ERROR- Bulk samples were received in the same shipping package as air samples for the same contaminant. Samples were not preserved or did not arrive at the laboratory within the recommended shipping time. Therefore, professional judgment must be used in the interpretation of results.
- 6- BLANK(s) NOT SUBMITTED- As required by the sampling and analytical method. Therefore, professional judgment must be used in the interpretation of results.
- 7- OTHER- Other laboratory specific comments requiring a disclaimer.

QUALITY ASSURANCE (QA)

The three CIHLs are accredited by the American Industrial Hygiene Association (AIHA) which requires participation in all applicable round robin testing programs. The AIHA accreditation program specifies operational guidelines for maintaining satisfactory performance, including qualified personnel, proficiency analytical testing, adequate facilities, quality controls, equipment maintenance, documentation and site audits. In addition to this accreditation program, all laboratories participate in several quality control programs for monitoring daily performance. Both internal and external quality control samples are analyzed to assure accuracy and precision of results. Some of the QA techniques used include replicate analyses, recycles, spiked controls, commercial reference controls, daily instrument calibration, control charts, regression analyses, data review, reagent and media blanks. Each laboratory maintains its own quality control manual, which gives extensive description of the quality assurance program. Please address specific QA questions to the CIHL performing the analytical work.

LAB ANALYTICAL EQUIPMENT

The primary analytical instrumentation in each laboratory consists of gas chromatographs, atomic absorption spectrophotometers (both flame and graphite furnace technologies), ultraviolet/visible spectrophotometers, infrared spectrophotometers, high performance liquid chromatographs, ion specific electrode meters, ion chromatographs, microbalances and microscopes (both phase and polarizing). Inductive coupled plasma (ICP) spectrometers, gas chromatograph/mass spectrophotometer, and an X-Ray Diffractometer are located at various labs. (See page 14 for applications and locations.)

SUBMISSION REQUIREMENTS

SAMPLE SUBMISSION FORM

Air samples must be submitted on Navy Environmental Health Center Forms NEHC 5100/13 and 5100/14. Copies of these form and instructions for completion are provided in the Industrial Hygiene Field Operations Manual, or may be requested from the laboratories.

BIOLOGICAL SAMPLES

Biological samples must be submitted with an accompanying sample submission documentation, preferably a transmittal list containing:

- 1- Name of medical facility submitting samples
- 2- Name of person submitting samples
- 3- Date of submission
- 4- Name of person sampled (i.e. patient)
- 5- Sample number [Last four digits of the social security number (SSN) of the patient, or CHCS number]. For example the sample number for J. Doe with SSN of 123-45-6789 might be #Doe 6789.
- 6- Age of person sampled (required for blood lead samples only)
- 7- Date sample was collected
- 8- Name of test requested.
- 9- Occupational code of patient
- 10- Patient's command UIC.

Because most medical treatment facilities use a computerized system for medical records, biological samples submitted for blood lead/ZPP and urine mercury may be submitted with a computerized transmittal list. Please refer to section below entitled "ROUTINE BIOLOGICAL SAMPLES" for specific guidance on this transmittal list.

Biological samples for blood lead/ZPP and urine mercury may be submitted on Standard Form 557 (miscellaneous Chemistry Chit). The request must be signed and dated by the submitting MD, RN or PA. All biological samples must be properly packaged and labeled in accordance with Navy, Federal, State and local regulations. It is recommended a commercial express package delivery service be used to transport samples to the CIHL. Please contact the carrier for their shipping and labeling requirements. In general, the samples must be placed in a sealed, waterproof primary container that contains absorbent material sufficient to absorb all possible leakage. The primary container must then be placed in a sealed, secondary container. The secondary container can then be placed in an outer container for shipment. All containers should be adequately cushioned so the samples do not become loose and move during shipment. Freezer packs should be used to keep the samples cold. Do not use ice, and do not freeze the samples. An Etiological Agent/Biomedical Material label must be affixed to the outside of the outer shipping container.

When samples are sent by U.S. Postal Service (USPS), Express Mail Delivery is required. Each package of samples using USPS cannot contain more than a total of 50 milliliters (1.7 ounces) of sample. If more than 50 milliliters of samples (e.g., approximately 7 blood lead samples) are sent to the lab, consider using a commercial express package delivery service. For more information on the shipment of samples, consult U.S. Postal Service Publication 52 entitled "Acceptance of Hazardous, Restricted, or Perishable Material" dated April 1990 and NAVSUPINST 4610.31A entitled "Preparation of Medical Material Requiring Freeze or Chill Environment for Shipment."

SAMPLING REQUIREMENTS

Always review the preferred method of sampling given in this guide and amplified by the appropriate analytical method (e.g., NIOSH or OSHA analytical method manuals, etc.). If the recommendation cannot be followed, contact the laboratory prior to sampling for additional guidance.

The recommended air volumes provided in this guide are usually a range of volumes, with the higher value recommended in the majority of sampling. The lower air volume should only be used when: 1) the exposure may be at an unsafe/unhealthful exposure level such as an exposure exceeding the Time Weighted Average (TWA) value given in the Occupational Safety and Health's Final Rule Limits, 2) the application of a Short Term Exposure Limit (STEL) or a Ceiling value is applicable to the substance, and 3) the operation limits the amount of sampling time. As a general rule, the recommended sampling volumes will allow a detection limit of 10-50% of the TWA.